

# **McIntyre Tailings Dam No. 3**

## **Laboratory Analysis**

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## 2.0 Materials and Methods

### 2.1 Sample collection sites

The three pits' locations were sampled on the beach of McIntyre Tailings area, west of Dam No. 3 on July 17, 2003 as given in Map 1. Pit 1 was approximately 20 meters from the current shoreline, Pit 2, 30 metres and Pit 3 in-between Pit 1 and 2, 10 meters north. The pits were dug with a shovel, making half a meter in diameter with a depth ranging from 0.6 to 0.9 m pits, exposing oxidised and un-oxidised tailings.

### 2.2 Sediment and Water sample collection and storage

Upon the completion of digging each pit, oxidised and un-oxidised tailings were scraped off the walls in each pit with the shovel and placed into plastic bags (10 kg each), tightly sealed. Table 1 below summarizes the tailings profiles from which a total of 5 samples were collected. One sample from Pit 1 (mixture of oxidized tailings), and two (oxidized and un-oxidized tailings) from Pit 2 and 3.

Table 1: Pit Layers Sampled

Pit	Sample	Tailings Description	Layer Depth (m)
1	1	Oxidized fine silt / fine sand	0.25 - 0.6
2	2	Oxidized fine sand	0.18 - 0.77
	3	Unoxidized tailings	0.77 - 0.85
3	4	Oxidized red + beige sandy clay	0.35 - 0.55
	5	Unoxidized tailings	0.55 - 0.63

Ten to fifteen minutes after each pit was dug, pore water infiltrating through the tailings was collected as 'grab' samples (in 250 ml polyethylene bottles with screw caps containing a foam liner) from the bottom of each pit.

Water samples were chemically analyzed (pH, Em, EC and temperature) in the field immediately after collection, are re-measured in the laboratory four days later. Also in the field, water samples were 1) filtered (through 0.45  $\mu$ m filter) and acidification with nitric acid and 2) acidified with

sulphuric acid in case a nutrient analysis will be later required. All samples were stored in a cooler with ice until arrival to the lab, followed by storage in the fridge at 4 °C.

Tailings samples were kept in the original plastic bags at room temperature (20 °C) until July 31, 2003 when chemistry was measured for the first time. After mixing the tailings thoroughly inside the bag, all four probes (pH, Em, EC) were submerged until readings were stable, usually within 5 seconds. After the measurements were completed, all probes were rinsed thoroughly with distilled water. The same technique was used for taking each slurry's supernatant water chemistry.

### 2.3 Slurry Dilution Preparation

Initially, one set of slurry dilutions (1:5) was prepared on July 31, 2003 for the 5 tailings samples. The chemistry for this set was measured 1 and 23 hours after the slurry preparation. On August 5<sup>th</sup>, 2003, three different slurry dilutions (1:1; 1:5 and 1:10) were prepared for each tailings sample collected from the 3 pits. The table below provides the proportions of tailings to distilled water for the slurry dilutions. These were prepared into 400 ml plastic beakers for the 1:1 and 1:5 ratio and in an 1L glass Erlenmeyer for the 1:10 dilution.

Table 2: Slurry information

Slurry Dilution	Tailings (g)	Distilled water (ml)
1 to 1	50	50
1 to 5	50	250
1 to 10	50	500

Following the addition of distilled water to each beaker, each slurry was stirred vigorously for a duration of 1 minute and left to settle for 1 hour before re-measuring the chemistry. Re-measurements were performed again after 24, 46, 70, 130, 230 and 310 hours. Beakers were covered with *parafilm* sheets through the entire experiment to prevent evaporation.

Four of the 1:10 dilutions slurries (Pit 2 oxidized, un-oxidized and Pit 3 oxidized and un-oxidized) were placed on an electronic stirring platform after the 1 hour measurement until the

7<sup>th</sup> day of the experiment, and 60 hours after decanting the supernatant water. Before re-measurements, these 4 samples were left to settle for 10 minutes.

On August 8, 2003 at 4:00 pm, (4 days after start up) the supernatant water was decanted for all 15 samples (3 dilutions) and stored in the fridge into centrifuge containers. Identical distilled water proportions were re added to the tailings in the beakers, followed by individual 1-minute vigorous stirring. The supernatant water chemistry was re-measured two more times; 2 days later, (100 hrs since decanting) and 5 days later.

On August 18, twenty-four samples were dispatched to Placer Dome's certified laboratory in Timmins for nutrient and element analysis. These include the initial (3) pore water samples, the supernatant water after decanting for the 3 dilutions (15 samples), the supernatant water for the 1:5 dilution after 24 hrs (5 samples) and Little Pearl Lake (LPL) grab water sample collected on July 16, 2003.

Table 3: Samples dispatched to Place Dome's Laboratory, Timmins

Sampling date	Stored up to	No. of samples	Description
17-Jul-03	19-Jul-03	3	Hole 1, 2 and 3 - Pore Water sampled from bottom of pits
31-Jul-03	1-Aug-03	5	(1:5 dilution) chemistry measured 1 hr after preparation, but sitting for ~ 1 day
5-Aug-03	8-Aug-03	15	(3 dillutions x 5 samples) decanted 70 hrs after preparing the slurries

## 2.4 Instrumentation and measurement procedure

The conductivity measurements were performed using an OAKTON Con 400 series EC instrument set to mS or  $\mu$ S mode that also reads temperature. The conductivity probe was calibrated for a conductivity range of 1.0 to 19,990  $\mu$ S at 25 °C, and wiped with isopropyl alcohol and rinsed with deionised water before the measurements of the experiment started. The probe was tested in standard solutions of 50, 500 and 10,000  $\mu$ S covering the range of conductivities encountered in the sample set.

A Corning 315 pH/Ion pH meter equipped with a combination electrode also by Coring #33221-034. The pH probe was calibrated with buffers at pH 4, 7 and 10 prior to pH determination.

Readings yielded after calibrating at pHs 4 and 7 were comparable to those of the 7 to 10; A sample measured after calibrating the pH probe between 7 to 10 reported the following values of 8.358 vs. 8.383 and 8.123 vs. 8.161, confirming that the higher pH values are indeed higher. A Corning M103 redox instrument with an inert platinum electrode and a standard Calomel probe was used to determine Em. The pH and Em probes were stored in saturated KCl solution.

### 3.0 Results

Table 4 describes the profiles of the 3 pits, and compares the tailings chemistry for the 5 collected samples (as stored) with the pits' bottom pore water chemistry measured first in the field and in the lab 4 days later.

The tailings' un-oxidized layers correspond to negative Eh and higher conductivity readings than the oxidized layer (-140 vs. 195 mV, Pit 2) and (2110 vs. 1720  $\mu$ S/cm, Pit 2) respectively.

The pore water pH's values increased as high as 1 unit from the initial field readings to those in the laboratory, 4 days after sampling. No significant changes are noted for redox or conductivity. The pH increase in the sample remains to be explained, as this also appears to take place in the tailings pond and the McSump seepage.

Table 4: Sediment vs. pore water (Field & Lab) chemistry

Pit	Sample	Sediment			Pore Water, Field			Pore Water - Lab				
		pH	Cond. ( $\mu$ S/cm)	Em (mV)	pH	Cond. ( $\mu$ S/cm)	Em (mV)	pH	Cond. ( $\mu$ S/cm)	Em (mV)	Alka. (mg/L)	Acid. (mg/L)
1	1	7.3	892	267	6.8	1220	46	7.7	1236	125	299	56
2	2	7.0	1720	195	6.7	2760	93	7.5	2970	97	386	152
	3	6.9	2110	-140								
3	4	6.9	1811	240	6.3	2800	113	7.4	3090	121	318	66
	5	6.9	2500	-158								

Table 5 compares the tailings' slurry dilution chemistry after 1 and 23 hours. After 23 hours the pH dropped very slightly, whereas the conductivity increased by 50% for all layers. Conductivity results demonstrate the tailings capability to release soluble elements to water over time.

Table 5: First slurry set (1:5) water chemistry after 1 and 23 hrs

Pit	Sample	1 hr					23 hrs		
		pH	Cond. ( $\mu\text{S/cm}$ )	Em (mV)	Alka. (mg/L)	Acid. (mg/L)	pH	Cond. ( $\mu\text{S/cm}$ )	Em (mV)
1	1	8.3	127	315	38.5	5.5	8.1	173	244
2	2	7.86	636	331	32	8.5	7.8	710	308
	3	7.97	371	223	40	7.6	7.6	490	270
3	4	7.95	410	240	28.3	18.4	7.8	428	271
	5	8.08	376	230	37.8	9.2	7.9	450	260

Tables 6a) to c) illustrates the changes in slurry chemistry after 3 days; every 24 hours. The pHs have remained constant throughout the experiment, for all layers of the pits. Figures 1a) to c) illustrate the pH variations over time for the 3 dilutions across the pits' layers.

The conductivity values after the 1<sup>st</sup> hour to 70 hours, increased for the 1:5 and 1:10 dilutions. However, these values were more stable for the 1:1 slurry dilution, as it is saturated. Figure 2a) to c) illustrates the conductivity results for the 3 pits for the oxidized and un-oxidized layers. Note that the 1:10 slurries's conductivity (for the 4 stirred samples) is comparable to that of the 1:5.

The redox readings remained relatively stable ranging from 188 to 317 mV in the 1:1 dilution, 200 to 309 mV (1:5) and 160 to 335 mV in the 1:10 dilution.

Table 6a: Slurry dilution (1:1) chemistry before decanting

Pit	Sample	1			24			46			70		
		pH	Cond. ( $\mu\text{S/cm}$ )	Eh (mV)	pH	Cond. ( $\mu\text{S/cm}$ )	Eh (mV)	pH	Cond. ( $\mu\text{S/cm}$ )	Eh (mV)	pH	Cond. ( $\mu\text{S/cm}$ )	Eh (mV)
1	1	7.8	592	468	8	667	492	8	1033	460	7.8	1059	431
2	2	7.5	1672	492	8	2380	509	7.8	2370	504	7.7	2400	434
	3	7.5	1114	484	7.6	1697	494	7.6	1310	506	7.3	1404	483
3	4	7.6	1122	482	7.7	1525	561	7.8	1200	488	7.6	1388	439
	5	7.6	1139	457	7.7	1474	545	7.8	1510	499	7.6	1654	440

Table 6b: Slurry dilution (1:5) chemistry before decanting

Pit	Sample	1 hr			24 hr			46 hr			70 hr		
		pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)
1	1	8.3	157	482	8.1	173	489	8.1	199	502	8	302	514
2	2	7.9	663	541	7.8	710	553	7.8	733	516	7.7	780	521
	3	7.8	453	515	7.6	490	514	7.6	575	488	7.4	600	508
3	4	8.1	390	529	7.9	428	516	7.9	508	499	7.7	661	526
	5	8	421	483	7.9	450	505	7.9	508	489	7.9	523	496

Table 6c: Slurry dilution (1:10) chemistry before decanting

Pit	Sample	1 hr			24 hr			46 hr			70 hr		
		pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)
1	1	8.6	90	500	8.3	300	410	8.3	124	502	8.1	140	523
2	2*	8.1	491	579	7.8	1291	466	7.8	1790	476	7.7	1808	506
	3*	8.3	185	433	7.7	590	403	7.7	614	444	7.6	688	511
3	4*	8.4	164	467	8	543	433	8	578	469	7.9	590	521
	5*	8.4	194	482	7.8	533	431	7.7	595	481	7.6	624	513

\* Stirred constantly after 1 hr measurement

Figure 1a: pH results for slurry (1:1) before decanting

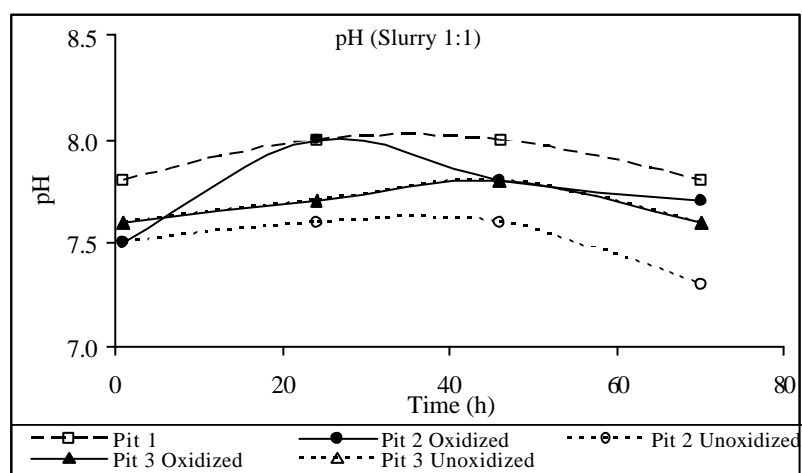




Figure 1b: pH results for slurry (1:5) before decanting

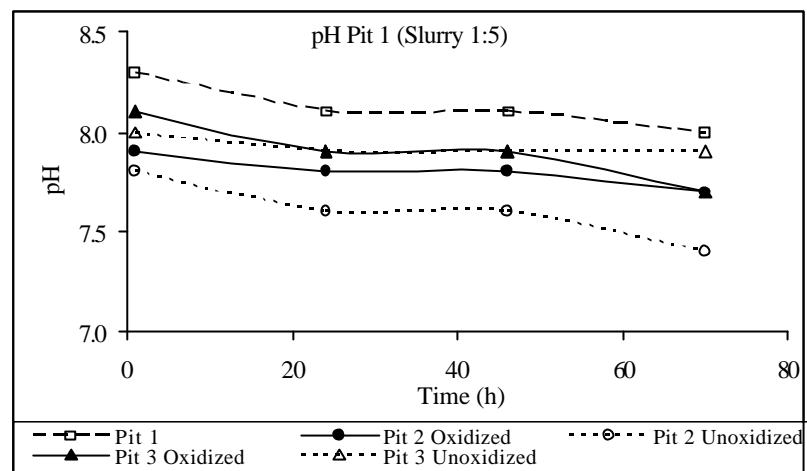


Figure 1c: pH results for slurry (1:10) before decanting

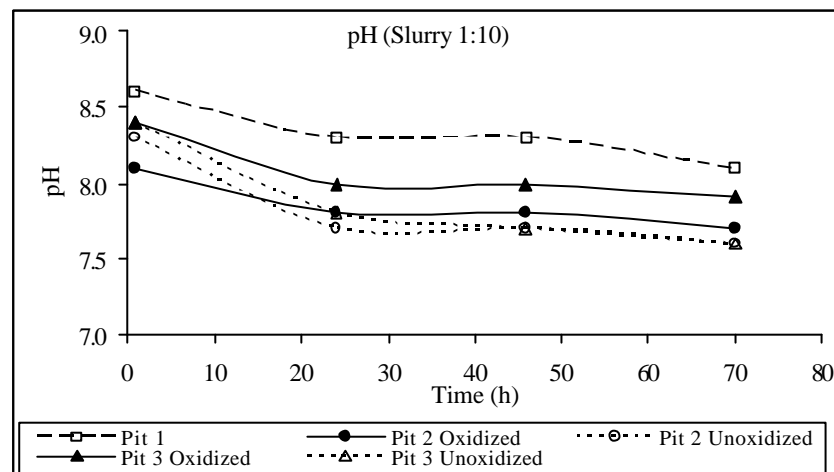


Figure 2a: Conductivity for slurry (1:1) before decanting

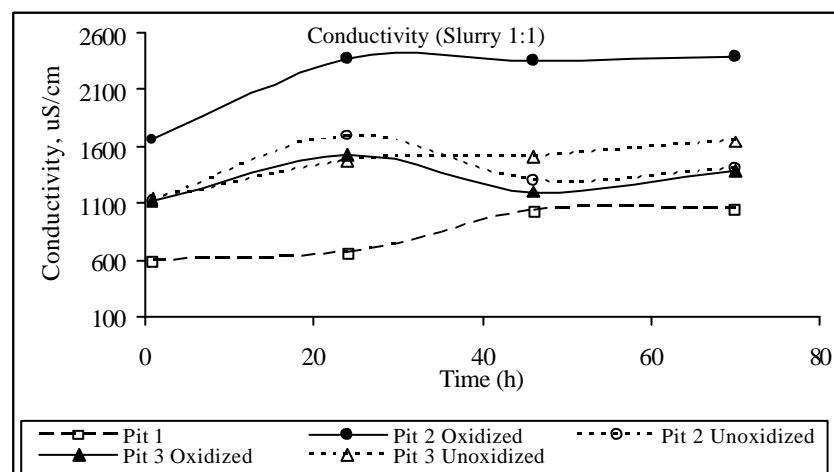


Figure 2b: Conductivity for slurry (1:5) before decanting

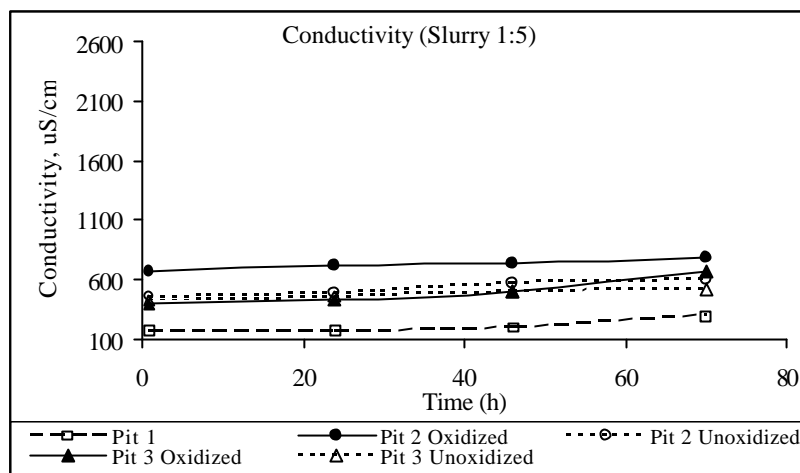
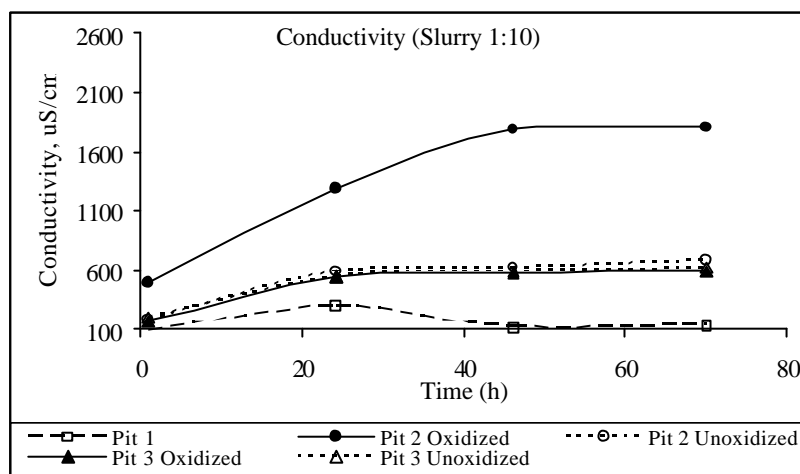


Figure 2c: Conductivity for slurry (1:10) before decanting



Tables 7 a) to c) display the slurry chemistry after the initial supernatant water was decanted, and replaced with fresh distilled water (same proportions). Values are given for 60, 180, 1236 and 4116 hours after decanting. These results suggest that there is a large pool of mobile elements in the pore water. Supernatant samples (15) were decanted and are presently with the PDPJA laboratory for ICP analysis.

Table 7a: Slurry dilution (1:1) chemistry after decanting and adding same volume distilled water to the remaining solids

Pit	Sample	60 hrs			180 hrs			1236 hrs			4116 hrs				
		pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	Acid. (mg/L)	Alkan (mg/L)
1	1	8.0	837	458	7.8	1126	413	7.8	916	456	7.9	1560	442	30.2	105
2	2	7.7	3730	452	7.9	3600	404	7.9	1557	465	7.9	1572	425	9.9	82
	3	7.5	1842	465	7.7	2130	381	7.7	1375	478	7.8	1765	414	10.9	84
3	4	7.7	2820	438	7.6	2840	436	7.2	1665	480	7.9	1629	399	9.9	89
	5	7.6	2290	448	7.6	2470	456	7.7	1399	488	7.9	1367	413	13.7	95

Table 7b: Slurry dilution (1:5) chemistry after decanting and adding same volume distilled water to the remaining solids

Pit	Sample	60 hrs			180 hrs			1236 hrs			4116 hrs				
		pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	Acid. (mg/L)	Alkan (mg/L)
1	1	8.1	380	517	8.1	174.5	407	8.2	391	430	8.0	830	401	7	89.8
2	2	7.9	1777	528	7.8	810	489	7.8	1262	445	7.7	1420	397	9.8	77.7
	3	7.8	920	554	8.0	445	455	7.9	601	449	7.8	871	399	9.5	86.4
3	4	7.9	892	467	7.9	488	488	7.9	630	450	7.8	823	400	7.9	100.8
	5	8.0	552	444	8.1	342	451	7.9	483	456	8.0	765	402	8.4	111.8

Table 7c: Slurry dilution (1:10) chemistry after decanting and adding same volume distilled water to the remaining solids

Pit	Sample	60 hrs			180 hrs			1236 hrs			4116 hrs				
		pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	pH	Cond. (μS/cm)	Eh (mV)	Acid. (mg/L)	Alkan (mg/L)
1	1	8.5	80	409	8.2	121	458	7.8	385	489	7.5	625	421	9	180.4
2	2	8.1	445	485	8.1	419	480	7.8	505	477	7.9	661	410	7.1	118.4
	3	7.8	373	469	7.8	365	490	7.6	437	472	7.5	693	404	13.6	144.9
3	4	8.1	140	423	8.0	186	484	8.0	378	449	7.4	652	396	9.1	168.9
	5	7.9	371	477	7.9	367	474	7.8	470	447	7.5	678	393	12.4	128.4

Figures 3 and 4 compare the slurry dilutions for Pits 2 and 3 (oxidized and un-oxidized layers), from the 1 measurement to the last, 4116 hours later. Sixty hours after decanting (130 hrs since 1<sup>st</sup> measurement) and the re-addition of distilled water, the pH slightly increased in the order-of-magnitude of the dilutions (highest for the 1:10). The conductivity response was different; the 1:1 dilution increased substantially in both oxidized and un-oxidized layers, but more so in the

oxidized. Before and after decanting the 1:1 slurry, the oxidized layer results are as follows: (2400 vs. 3730  $\mu\text{S}/\text{cm}$ ) in Pit 2 and (1388 vs. 2820  $\text{S}/\text{cm}$ ) in Pit 3.

Ten days after decanting, overall the conductivity decreased for the 1:5 and 1:10, however it oscillated for the 1:1 dilution, reaching peak values on the 310<sup>th</sup> day. The pH remained steady with the exception of the 1:1 dilution, un-oxidized layer where the pH dropped from 7.7 to 7.0 and back to 7.7 for the 130, 230 and 310 hours of re measurements.

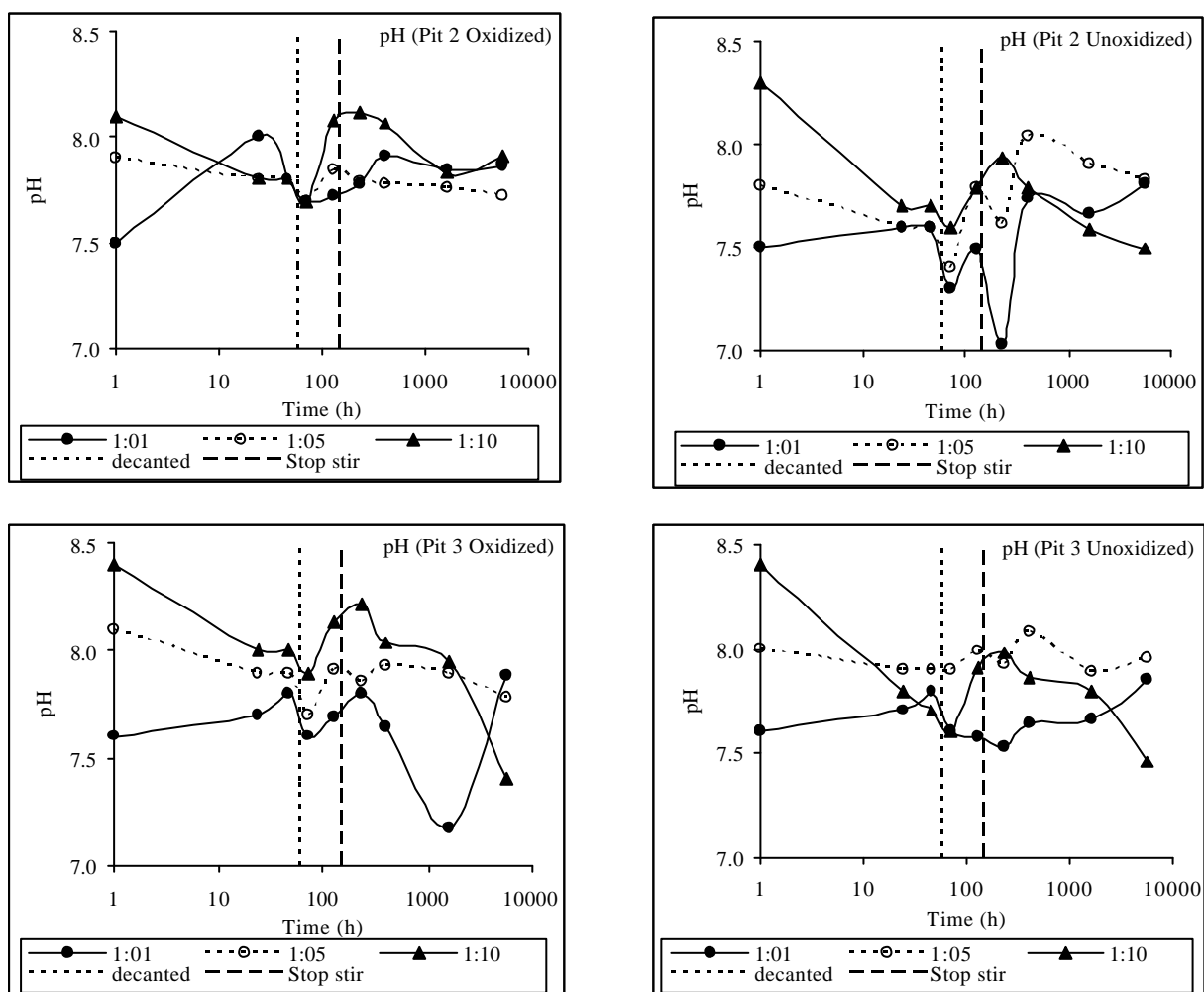


Figure 3: pH change comparison for the 1:1, 1:5, and 1:10 dilutions, oxidized vs. Unoxidized layers

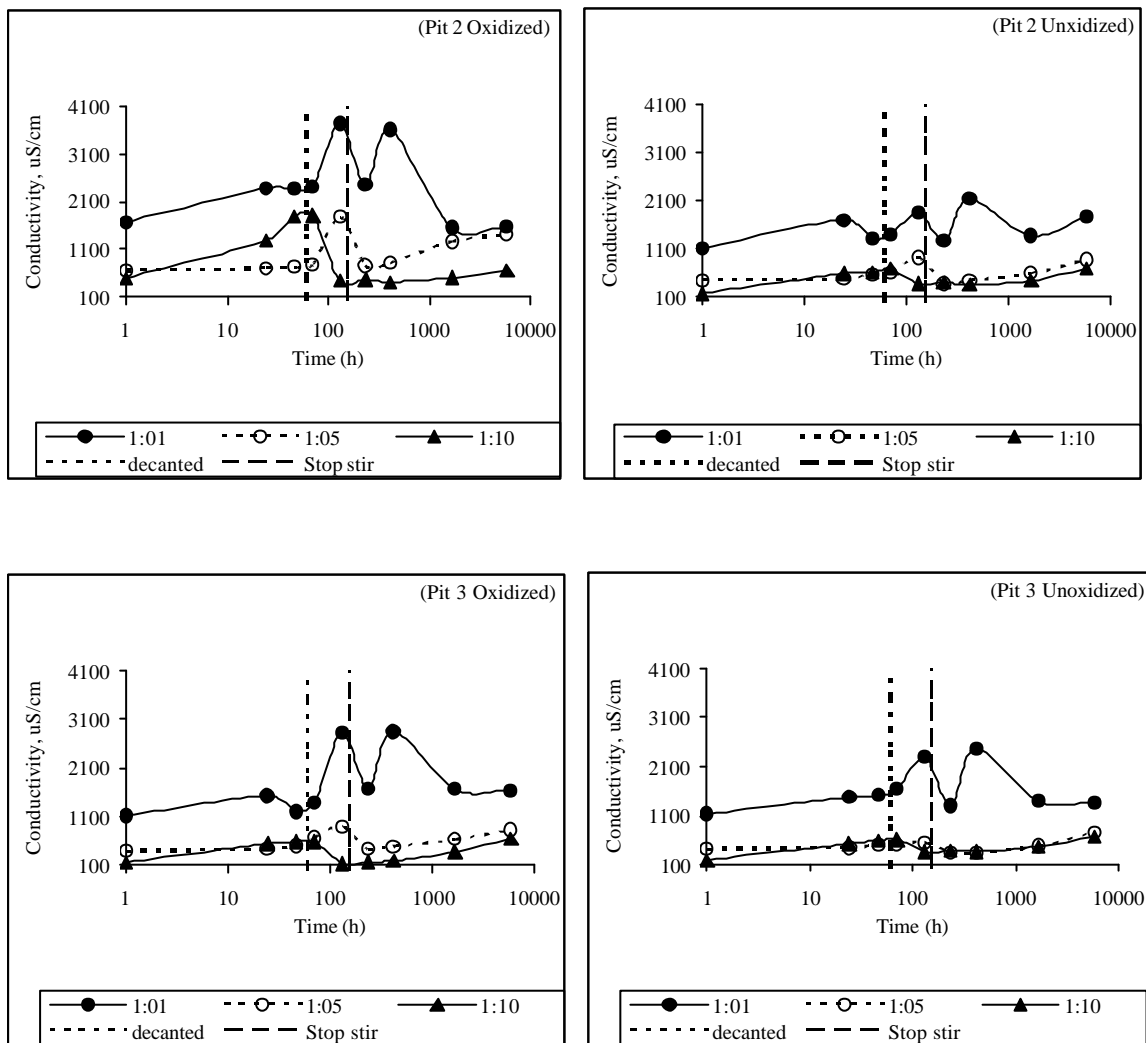


Figure 4: Conductivity change comparison for the 1:1, 1:5, and 1:10 dilutions, oxidized vs. Unoxidized layers

Table 8: Effect of stirring on water chemistry of the slurries with dilution ratio 1:1, 1:5 and 1:10

Pit	Sample	pH	Cond. (µS/cm)	Eh (mV)	pH	Cond. (µS/cm)	Eh (mV)
		Before stirring			After stirring		
1:1 slurry							
1	1	7.9	1560	442	7.6	1342	393
2	2	7.9	1572	425	7.8	1224	392
	3	7.8	1765	414	7.5	1657	380
3	4	7.9	1629	399	7.6	1516	397
	5	7.9	1367	413	7.7	1545	347
1:5 slurry							
1	1	8.0	830	401	7.7	820	430
2	2	7.7	1420	397	7.5	1408	476
	3	7.8	871	399	7.7	880	365
3	4	7.8	823	400	7.7	814	436
	5	8.0	765	402	7.7	681	366
1:10 slurry							
1	1	7.5	625	421	7.5	619	413
2	2	7.9	661	410	7.8	659	432
	3	7.5	693	404	7.6	698	405
3	4	7.4	652	396	7.5	670	430
	5	7.5	678	393	7.6	705	413

Table 9: Water chemistry of the samples from ERG site

Samples	20-Aug-03					28-Jan-04									
	Without stirring					Before stirring					After stirring				
	pH	Cond. (µS/cm)	Em (mV)	Acid. (mg/L)	Alkan. (mg/L)	pH	Cond. (µS/cm)	Em (mV)	Acid. (mg/L)	Alkan. (mg/L)	pH	Cond. (µS/cm)	Em (mV)	Acid. (mg/L)	Alkan. (mg/L)
ERG	2.9	1874	557	311.9	-	3.0	1639	443	331.0	-	-	-	-	-	-
Mess behind head from Gilles Lake	2.7	2020	349	283.5	-	4.8	1490	280	49.9	0	-	-	-	-	-
Mine discharge sludge little Pearl Lake	7.1	2490	403	183.6	422.7	7.1	2410	148	24.0	312.9	7.0	2188	201	-	-